
COMBUSTION, EXPLOSION,
AND SHOCK WAVES

Influence of the Dispersity of Aluminum Powder on the Ignition Characteristics of Composite Formulations by Laser Radiation

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Abstract—The results of an experimental study of the regularities of the ignition of composite formulations containing aluminum powders of different dispersity by a CO₂ laser are presented. The dependences of the ignition delay time and thermal mode on the irradiance and the composition of the composite formulation are obtained.

Keywords: composite formulations, laser radiation, aluminum powder dispersity, ignition delay, thermal mode.

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INTRODUCTION

The regularities of the radiant ignition of composite formulations (CFs) are of interest for the theory of the thermal ignition of condensed heterogeneous formulations of complicated structure [1] and have an obvious practical significance in developing ignition systems and in assessing possible unauthorized ignition of solid propellant motors and gas generators.

In the last decade, interest in the use of ultradispersed powders (UDPs) of metals in high-energy fuel compositions has increased sharply [2]. This is due to the fact that traditional CFs, containing 5- to 15- μm commercial aluminum powders, completely exhausted their potential, while the progress in the technology of metal UDPs with an average particle size of 0.1 μm (in particular, electric explosion of wires) allows producing large quantities of metal powders with controllable properties and high stability [3]. It was shown [4–11] that replacing traditional aluminum powders by UDPs can increase the burning rate of composite formulations by more than 2.5 times, decrease the pressure exponent in the burning rate law, and reduce the ignition delay time. In a number of studies, an abnormal (explosive) burning of energetic composite formulations containing aluminum UDP was observed [12]. The main reason for anomalous phenomena is a high chemical activity of metal UDPs. It is known that an increase in the specific surface area of metal powders is accompanied by an increase in their pyrophoricity. Therefore, the use of UDPs as energetic additives entails increased fire and explosion hazard in the production

and handling of CFs. Knowledge of the basic laws governing the behavior of metal UDPs in CFs will reduce the probability of occurrence of abnormal phenomena, extend the scope of practicable nanosized metal additives, and thereby significantly improve the performance of composite formulations.

In recent years, a number of works on the ignition of CFs containing metal UDPs have been published. In [6, 7], the effect of dispersity of aluminum and boron powders with an average particle size of 0.1 μm on the ignition of CFs unfiltered radiant flux from a 5 kW xenon lamp was studied. Composite formulations based on ammonium perchlorate (AP), HMX, and nitrile rubber (NBR) were examined. The content of aluminum powder was 25 wt %. The experiments demonstrated that a partial or total replacement of ASD commercial powders by Alex UDP in CFs leads to a 1.5- to 2.2-fold decrease in the ignition delay at irradiances of 30 to 110 W/cm². In [8–10], experiments on the ignition of composite formulations prepared from ammonium perchlorate, hydroxyl-terminated polybutadiene (HTPB), and 15 wt % aluminum powder by a CO₂ laser showed that, with increasing aluminum powder dispersity, the ignition delay time decreases at irradiances of 90–600 W/cm² and pressures of 0.1–1.0 MPa. At the same time, in [11], no influence of 5 wt % aluminum UDP on the characteristics of the radiant ignition of an AP-based CF was observed. The authors of [11] showed that Fe₂O₃ powder most strongly influences the ignition of CFs.

This paper presents the results of an experimental study of the characteristics of ignition and thermal

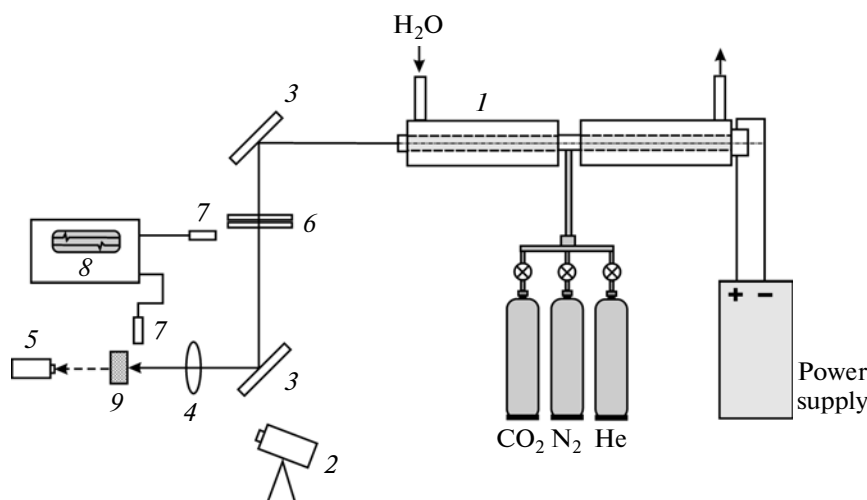


Fig. 1. Schematic of the experimental setup (see the text).

mode for composite formulations containing aluminum powder of different dispersities.

EXPERIMENTAL

The CF samples were ignited using a laser setup assembled around a multimode cw CO₂ laser with a wavelength of 10.6 μm and a maximum power of 100 W (Fig. 1). Upon opening central type shutter 6, the beam (20 mm in diameter) of CO₂ laser 1 was focused with a system of mirrors 3 and sodium chloride lens 4 onto sample 9. The ignition delay time was determined using the signals from two photodiodes 7, one of which triggered memory oscilloscope 8 upon shutter opening, whereas the second recorded the appearance of a flame over the sample. The power of laser radiation incident on the CF sample was measured with IMO-2 radiation power meter 5. The time of shutter opening did not exceed 0.5 ms. The duration of the laser pulse was varied within 30–1500 ms, being determined from the condition of secure ignition and subsequent burning of the CF sample. The temperature field on the surface of the CF sample during laser ignition was recorded at a wavelength of 2.5 to 2.7 μm using a Jade J530 SB digital thermal imager. The imager was equipped with lenses with focal lengths of 25, 50, and 200 mm. The optical axis of the imager was directed onto the sample surface at an angle of 45°. The distance from the CF sample to the camera was 0.8 m. Imaging was performed at a frequency of 100 Hz. The error in measuring the temperature with the imager was 5%. The relative error in measuring the ignition delay time t_{ign} at a confidence level of 95% was 8–18%. The main error in determination of the radiation power was 5%.

COMPOSITIONS OF THE TEST COMPOSITE FORMULATIONS

The effect of the dispersity of aluminum powder on the characteristics of inflammation was investigated for two basic solid formulations. The first formulation (formulation A) contained 24 wt % grade MPWT-LD energetic binder, 56 wt % mixed oxidizer oxidizer (50 : 50 ammonium nitrate–HMX) and 20 wt % aluminum powder. The second formulation (formulation B) contained 12 wt % SKDM-80 inert rubber, 73 wt % mixed oxidizer (40 : 40 : 20 ammonium nitrate–HMX–ammonium perchlorate) and 15 wt % aluminum powder. The coefficient of oxidizer excess for these compositions was 0.545. The metallic fuel was ASD-4 aluminum powder (with a weight-average particle diameter of $D_{43} = 3.5 \mu\text{m}$) and Alex ($D_{43} = 0.15 \mu\text{m}$) prepared by electrical explosion of conductors [13]. For each of the basic compositions of CFs, the ratio between the contents of ASD-4 and Alex aluminum powder was varied. The dispersity of ammonium nitrate (AN), HMX, and ammonium perchlorate powders was 160–315 microns. The compositions of the composite formulations are given in Table 1. CF samples of cylindrical shape with a diameter of 10 mm and a height of 30 mm were prepared by mechanical mixing of initial components followed by die or static pressing at ~215 MPa and curing. The density of the cured samples was $\rho = 1.4\text{--}1.65 \text{ g/cm}^3$, depending on the composition. In duplicate experiments for the same compositions, the scatter in the sample density was within 0.02 g/cm^3 . The porosity of the samples was not controlled, being considered insignificant. Immediately before the experiment, the irradiated surface of the sample was prepared by cutting with a microtome knife. The sample height was 5 mm. In this case, the end face surface was smooth without dents and protrusions. To reduce optical heterogeneity, the end face surface of the test CF was covered with lamp soot.

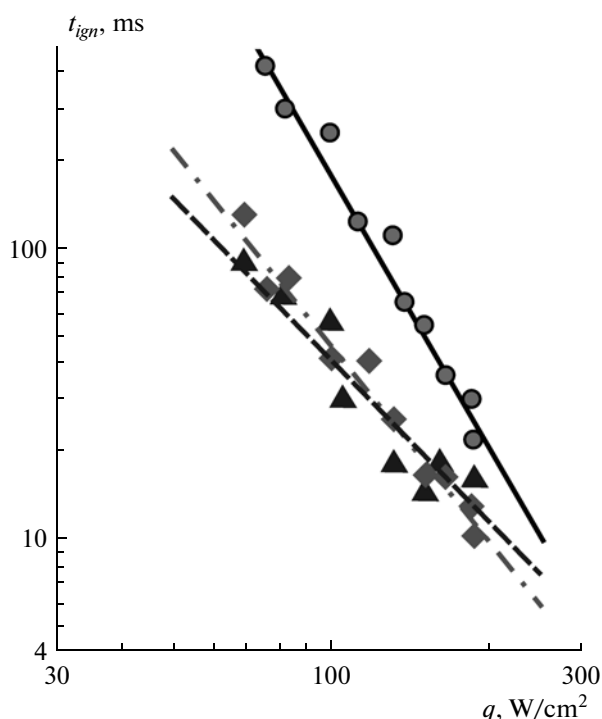


Fig. 2. Ignition delay time for compositions A1–A3 (●, ▲, ◆) as a function of the laser irradiance.

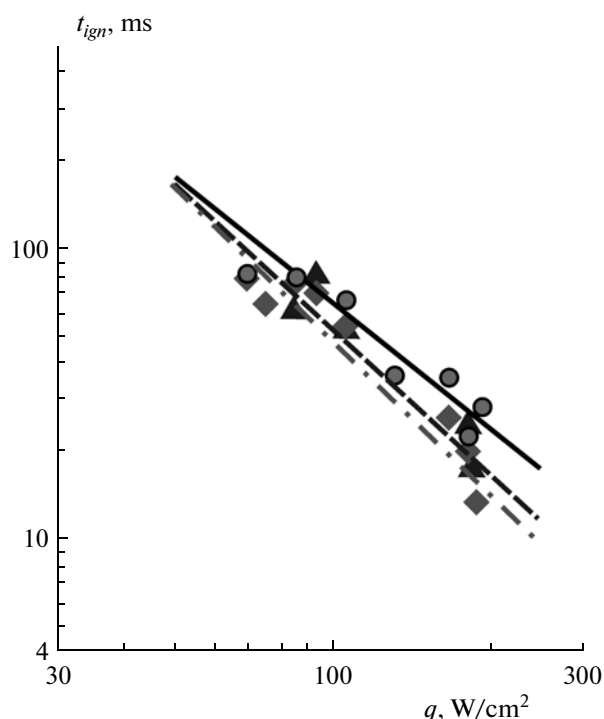


Fig. 3. Ignition delay time for compositions B1–B3 (●, ▲, ◆) as a function of the laser irradiance.

EXPERIMENTAL RESULTS

Thus, we obtained the dependences of the ignition delay time t_{ign} of the test CFs containing aluminum powders of different dispersities on the irradiance. Some of the results, averaged over three duplicate experiments for each value of q , are displayed in Figs. 2 and 3. In all the experiments, we observed stable ignition and burning of the CF samples.

Within $q = 60\text{--}200\text{ W/cm}^2$, the experimental data were approximated, using the least-squares method, by the functional dependence

$$t_{ign} = bq^{-n}, \quad (1)$$

where $[t_{ign}] = \text{ms}$, $[q] = \text{W/cm}^2$. The obtained values of the parameters b and n are given in Table 2. In all the

experiments, the ignition delay time of CFs decreased with increasing irradiance.

The effectiveness of the influence of the type of fuel-binder and oxidizer on the ignition of CFs was characterized by the ratio of the ignition delay time of basic formulation A, composed of AN–HMX binary oxidizer and MPVT-LD active binder, to that of formulation B, consisting of AN–HMX–AP ternary oxidizer and SKDM-80 inert binder, at the same dispersity of the aluminum powder component. For example, at $q = 75$ to 190 W/cm^2 , the ignition delay time for formulation A1 is 4.7–0.8 times higher than that for formulation B1 (containing ASD-4); for formulation A2, the ignition delay time is 0.9 times that for formulation B2 (containing aluminum powder ASD-4 and Alex); for formulation A3, the ignition delay time is

Table 1. Compositions of composite formulation

Composition	Content of components, wt %						
	AN	HMX	AP	MPVT-LD	SKDM-80	Alex	ASD-4
A1	28	28	—	24	—	—	20
A2	28	28	—	24	—	10	10
A3	28	28	—	24	—	20	—
B1	29	29	15	—	12	—	15
B2	29	29	15	—	12	5	10
B3	29	29	15	—	12	15	—

1.1–0.8 times that for formulation B3, containing Alex aluminum UDP. Thus, at low irradiances, the influence of the type of fuel-binder and oxidizer of composite formulations containing ASD-4 micron-sized aluminum powder is more significant than at higher irradiances. Partial or complete replacement of ASD-4 micron-sized aluminum powder by Alex ultrafine powder in CFs reduces the ignition delay time under the same conditions of ignition.

The influence of the dispersity of aluminum powder on the ignition of CFs was characterized by the efficiency coefficient K , which was defined as the ratio of the ignition delay time for the basic CF, containing ASD-4 (A1 or B1), to that for a CF containing Alex aluminum UDP. The results for the influence of the aluminum powder dispersity on the ignition of CFs are presented in Tables 3, 4. With increasing aluminum powder dispersity in basic formulation A, its ignitability improves at low and moderate irradiances. The ignition delay time for formulations containing aluminum UDP is 4–6 times lower than that for formulation A1 with micron-sized aluminum powder. At higher intensity of heat radiation, the efficiency of influence of the aluminum powder dispersity is lower ($K = 1.3–2.4$). For basic formulation B, the efficiency of the influence of the aluminum powder dispersity is negligible ($K = 1.0–2.4$) compared to basic formulation A. At low and moderate irradiances, the aluminum powder dispersity does not affect the ignition delay time, whereas at 190 W/cm², the aluminum powder dispersity produces a significant effect. Thus, the introduction of aluminum UDP has the most pronounced impact on reducing the ignition delay for formulation A at $q < 150$ W/cm² and for formulation B at $q > 150$ W/cm².

Using a Jade J 530 SB thermal imager, we measured the burning surface temperature and monitored the dynamics of the ignition of basic formulation A at an irradiance of 60 W/cm². Figures 4–8 show typical thermograms and thermal images of the ignition and burning of basic formulation A. Formulation A3, based on MPVT-LD energetic binder and containing 20 wt % aluminum UDP, ignited at ~580–610°C (Fig. 4). The temperature distribution over the burning surface of composite formulation A3 at the moment of emergence of a visible flame is shown in Fig. 5 (r is the radial coordinate of the sample). The burning of formulation A3 occurs features significant variations in the surface temperature, within 480–740°C. This is likely to be a consequence of an intense heat transfer from the burning surface of the sample into the condensed phase and agglomerated aluminum UDP particles and combustion products (Fig. 6). Formulation A1, containing 20 wt % ASD-4 aluminum powder, ignites at substantially higher temperatures, ~530–820°C (Fig. 7). Figure 8 shows the temperature distribution over the burning surface of the A1 sample at the moment appearance of a visible flame (r is the radial coordinate of the sample). A ther-

Table 2. Values of b and n in dependence (1) at $q = 60–200$ W/cm²

Composition	$b \cdot 10^{-6}$	n
A1	375	3.16
A2	0.25	1.89
A3	1.59	2.27
B1	0.04	1.42
B2	0.11	1.66
B3	0.17	1.77

Table 3. Efficiency of aluminum UDP additive on the ignition of formulations A2 and A3

q , W/cm ²	t_{ign} , ms (formulation A1)	K	
		formulation A3	formulation A2
75	425 ± 25	6.0	6.0
100	250 ± 20	6.1	4.5
130	110 ± 20	4.4	6.1
150	54 ± 10	3.4	3.9
190	21 ± 3	2.1	1.3

Table 4. Efficiency of aluminum UDP additive on the ignition of formulations B2 and B3

q , W/cm ²	t_{ign} , ms (formulation B1)	K	
		formulation B3	formulation B2
75	80 ± 5	1.3	1.3
100	72 ± 8	1.3	1.4
130	36 ± 2	1.0	1.1
150	35 ± 2	1.3	1.3
190	28 ± 3	2.1	1.6

mal imaging cinematography of the ignition of formulation A1 showed that, in the initial period, a faint glow of the combustion products and the heat-up of the sample surface are observed, with the burning surface temperature increasing from 460 to 620°C. The reaction layer is supposedly thicker than that for formulation A3, so that the chemical reaction, accompanied by the release of heat and decomposition products, occurs in deeper layers. When the temperature reaches ~620°C, an intense release of heat and degradation products occur, accompanied by the ejection of the surface layer of the condensed phase. As a result, the temperature of the bare surface of the sample increases sharply to ~820°C due to the heat accumulated in the deeper layers.

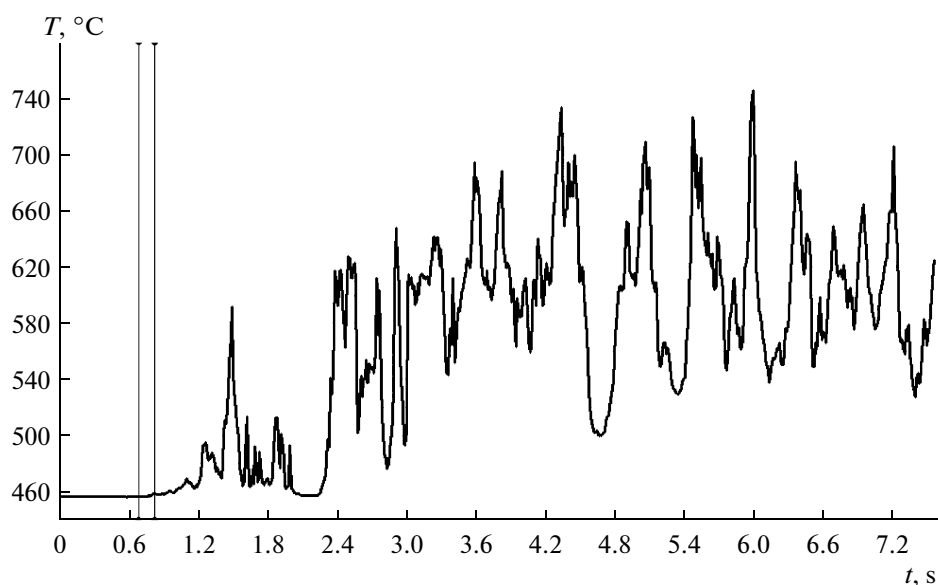


Fig. 4. Thermogram of the ignition and burning of composite formulation A3.

Formulation A1 burns at surface temperatures of 540–720°C. Thus, the ignition of formulation A3, containing Alex ultrafine aluminum powder, occurs on the surface of the sample at 580–610°C. The ignition of formulation A1, containing ASD-4 micron-sized aluminum powder, occurs in deep layers of the condensed phase of the sample at 530–820°C. In this case, the time of reaction layer heating and ignition is much longer than that for formulation A1 at an irradiance of 60 W/cm².

KINETIC CONSTANTS OF IGNITION

The mathematical formulation of the problem of ignition of a condensed opaque material by a radiant heat flux is reduced to a quasi-linear one-dimensional

heat conduction equation with the appropriate boundary and initial conditions [1]:

$$\frac{\partial T}{\partial t} = a \frac{\partial^2 T}{\partial x^2} + \frac{Qz}{c} \exp\left(-\frac{E}{RT}\right), \quad (2)$$

$$T(x, 0) = T_0 = \text{const} \quad \text{at} \quad 0 \leq x < \infty,$$

$$-\lambda \frac{\partial T}{\partial x} = q_s = \text{const} \quad \text{at} \quad x = 0, t > 0, \quad (3)$$

$$\frac{\partial T}{\partial x} = 0 \quad \text{at} \quad x \rightarrow \infty, t > 0.$$

Here, T is the temperature; a , λ , and c are the thermal diffusivity, thermal conductivity and specific heat of the condensed material; Q is the specific (per unit mass) heat of reaction; z is the preexponential factor; E is the activation energy; R is the universal gas constant; and q_s is the absorbed (with consideration given to reflection) fraction of the radiant flux.

The theory of ignition by radiant energy allows solving the inverse problem: the definition of the formal macrokinetics of ignition. The results of the numerical solution of boundary problem (2), (3) with error less than 6% can be approximated by the formula [1]

$$t_{ign} = 0.35 \left(1 - \frac{T}{T_*}\right)^2 \frac{Ec}{RQz} \exp\left(\frac{E}{RT_*}\right). \quad (4)$$

As the temperature scale, we used the steady-state temperature of ignition T_* , which is determined by equating the rates of heat release by the chemical reac-

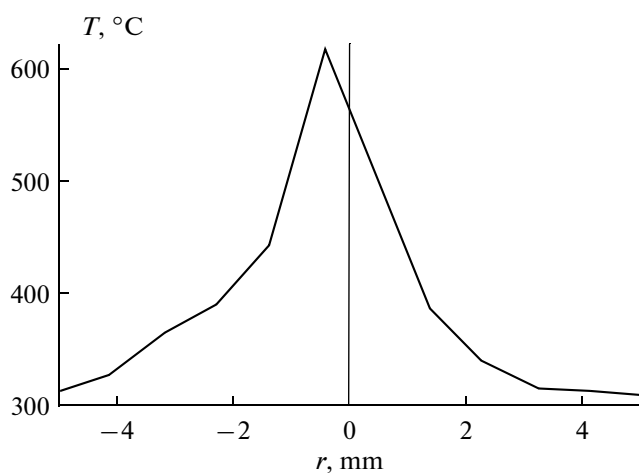


Fig. 5. Profile of the surface temperature of formulation A3 at the moment of ignition ($t_{ign} = 140$ ms).

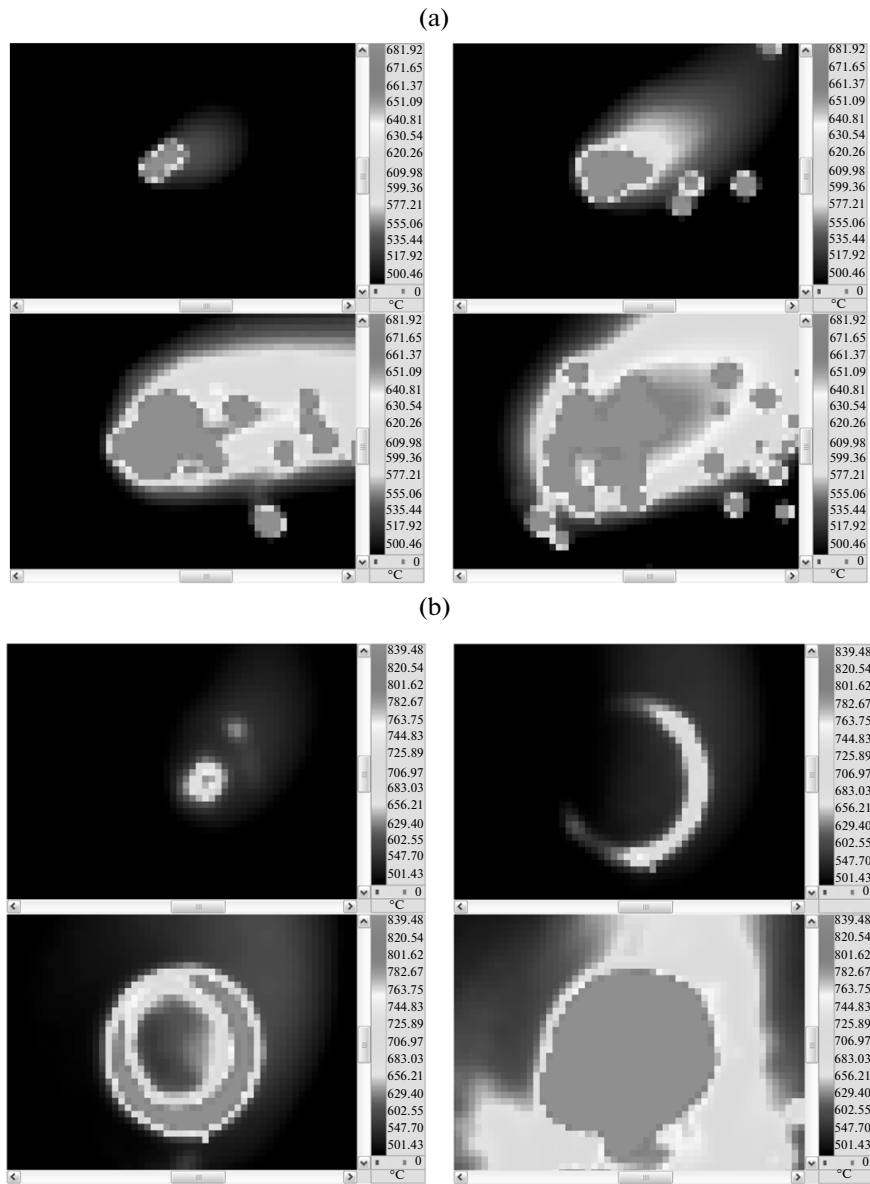


Fig. 6. Cinematographic frames of thermal imaging of (a) formulation A3 at time intervals of $\Delta t = 0.14$ s and (b) formulation A1 at time intervals of $\Delta t = 2$ s.

tions in the reaction layer and of heat transfer into the condensed phase [1]:

$$T_* = T_0 + 1.2q_s \left(\frac{t_{ign}}{\lambda c \rho} \right)^{1/2}. \quad (5)$$

Expression (4) can be recast as

$$\ln \frac{t_{ign}}{(1 - T/T_*)^2} = \ln \frac{0.35Ec}{RQz} + \frac{E}{RT_*}. \quad (6)$$

In the $\ln \frac{t_{ign}}{(1 - T/T_*)^2}$ versus $\frac{1}{T_*}$ coordinates, this equation is represented by a straight line, the slope of

which yields the effective activation energy and, therefore, at known thermophysical constants, the product Qz (or preexponential factor z). Thus, the experimental dependences of the ignition delay time on the heat flux in conjunction with equations (5), (6) make it possible to determine the constants of the formal kinetics of the ignition process.

The kinetic constants of the ignition of the CFs were calculated without regard for the reflection coefficient at the following thermophysical parameters: $\rho = 1.87$ g/cm³, $c = 1.24$ kJ/(kg K), and $\lambda = 0.66$ W/(m K).

The determined values of the activation energy and preexponential factor for the formulations based on the ternary oxidizer (AN–HMX–AP) and SKDM-80 inert binder are presented in Table 5. For the formula-

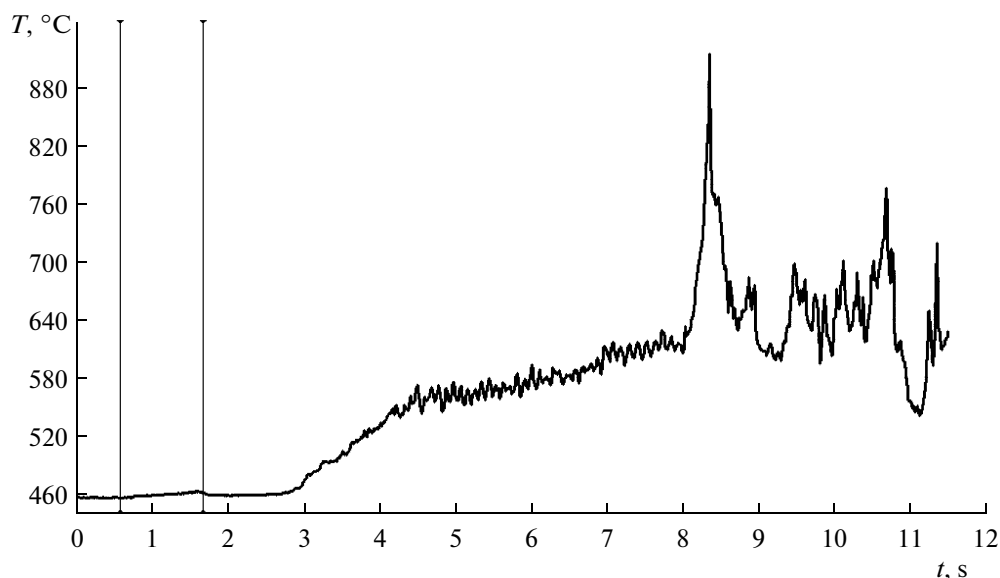


Fig. 7. Thermogram of the ignition and burning of composite formulation A1.

tions based on the binary oxidizer (AN–HMX) and MPVT LD energetic binder, the exponent of approximation function (1) is $n > 2$ (Table 2), indicative of an increased role of the gas phase in the process of ignition. Therefore, these data were not processed according to the above procedure, since it is based on a solid-phase model of ignition [1].

ANALYSIS OF RESULTS

Analysis of the results of studying the effectiveness of the influence of the type of energetic binder and the aluminum powder dispersity on the ignition of CFs showed that, with increasing irradiance, the effect of the aluminum powder dispersity reduces for basic for-

mulation A, prepared from AN–HMX oxidizer and MPVT-LD energetic binder, whereas for basic formulation B, composed of AN–AP–HMX ternary oxidizer and SKDM-80 inert binder, it increases. Apparently, this is associated with the thermal stability of the energetic binder used, a temperature increase in the reaction layer of the condensed phase, and a growing role of the gas phase in the ignition of the composite formulation. Increasing the dispersity of aluminum powder in the composite formulation influences the rates of the respective chemical reactions and the amount of heat supplied to the condensed phase. This is evidenced by the results on the thermal decomposition of mixtures of ammonium nitrate and HMX with metal UDP additives, as well as by a weaker pressure dependence of the burning rate for CFs containing aluminum UDP [14]. Addition of UDP aluminum to CFs leads to an increase in the heat release rate because of the combustion in a zone close to the burning surface (condensed phase) and, hence, a more rapid heating of the subsurface reaction layer of the formulation due to a more intense conduction heat transfer at the expense of UDP aluminum particles.

Analysis of the results of thermal imaging cinematography showed that the radiant heat flux reaches the surface of the composite formulation, part of which is absorbed whereas the other is reflected. The amount of absorbed radiant heat flux depends on the composition of the CF and the radiation flux wavelength. It should be noted that the components of a composite formulation (fuel-binder, particles of oxidizer and aluminum) have different refractive indices, which leads to a difference in the energies absorbed by the components in the initial stage of ignition. As a result, the heating of the AN, HMX, and AP particles may be delayed, whereas the fuel-binder will heat the adjacent

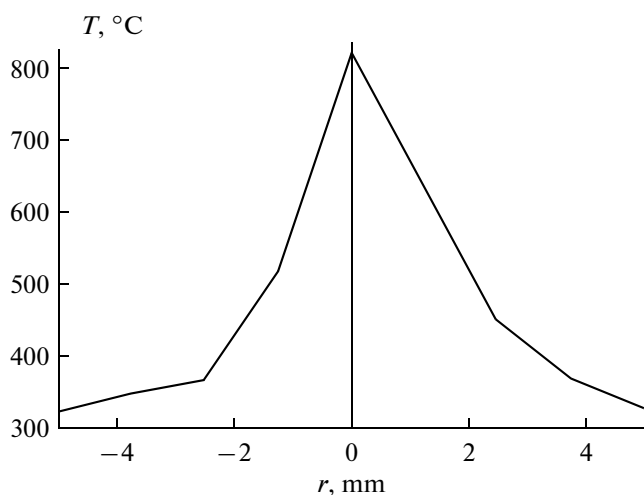


Fig. 8. Profile of the surface temperature of formulation A1 at the moment of ignition ($t_{ign} = 1140$ ms).

Table 5. Values of the scale temperature and formal kinetics constants for formulations B1–B3 at $q = 60\text{--}200\text{ W/cm}^2$

Composition	T_* , K	E , kJ/mol	Q_z , W/g
B1	500–586	78.4	$8.89 \cdot 10^{11}$
B2	493–538	147	$3.85 \cdot 10^{19}$
B3	490–520	226	$1.85 \cdot 10^{28}$

oxidizer and aluminum particles through heat conduction. The heating rate and the thickness of the reaction layer of the condensed phase will depend on the particle size of the oxidizer and aluminum. During the period of ignition delay, bubbles of partially decomposed fuel-binder are formed on the CF sample surface. Apparently, this is because the absorption of thermal radiation by deep layers of the CF and heat removal by conduction to the environment results in the maximum temperature of the condensed phase being reached somewhere below the surface of the fuel. The formation of bubbles in the fuel-binder occurs under conditions that provide a relatively long ignition delay (low radiant heat flux), being most intense for MPVT LD energetic binder (with low thermal stability). There is a possibility that the radiant heat flux, passing through the oxidizer crystals, can give rise to local hotspots in subsurface layers of the fuel, thereby greatly increasing the likelihood of reactions in these layers during the ignition process. As a result, the temperature in the subsurface layer can be much higher than the surface temperature of the condensed phase. The gas phase can also influence the ignition of CFs. If the process of ignition is dominated by heterogeneous reactions, the influence of the environmental is negligible. However, if an essential factor in the ignition process is gas-phase reactions (at low irradiances), the influence of the environment increases due to a lowering of the temperature and the rate of chemical reactions.

CONCLUSIONS

(1) Partial or complete replacement of ASD-4 micron-sized aluminum powder by Alex ultrafine powder in the composition of the studied composite formulations reduces the ignition delay time 1.3- to 6.0-fold at irradiances of $q = 60\text{--}200\text{ W/cm}^2$ under identical conditions of ignition.

(2) The influence of the type of fuel-binder (active or inert) on reducing the ignition delay time in composite formulations based on ASD-4 micron-sized aluminum powder is more significant at low irradiances ($q < 150\text{ W/cm}^2$).

(3) The effect of aluminum ultrafine powder on shortening the ignition delay for the composite formulations composed of AN–HMX binary oxidizer and

MPVT-LD energetic binder is more significant at low irradiances ($q < 150\text{ W/cm}^2$), whereas for composite formulations based on AN–AP–HMX ternary oxidizer and SKDM-80 inert fuel-binder, at $q > 150\text{ W/cm}^2$.

(4) The ignition of CFs based on AN–HMX binary oxidizer and MPVT-LD energetic binder and containing Alex ultrafine aluminum powder occurs in the condensed phase on the sample surface at temperatures of $\sim 580\text{--}610^\circ\text{C}$. The ignition of CFs containing ASD-4 micron-sized aluminum powder occurs in subsurface layers of the condensed phase of the sample at $530\text{--}820^\circ\text{C}$, with the time of heating of the reaction layer and ignition delay time being 6-fold longer at an irradiance of 60 W/cm^2 .

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